### Phase I

### Monthly Progress Report

Sept. 3, 1996 Report No. 5

Reporting Period: August 3 - Sept. 3, 1996

## **CONTRACT TITLE AND NUMBER:**

"Advanced Monitoring of Groundwater Cleanup Technologies" - Phase I F41624-96-C-0006

### **CONTRACTOR NAME:**

Advanced Fuel Research, Inc. 87 Church Street
East Hartford, CT 06108

### **KEY PERSONS:**

James R. Markham Chad M. Nelson

### **CONTRACT PERIOD:**

April 19, 1996 - October 4, 1996 (Extended to November 29, 1996)

#### DUE DATE OF PHASE II PROPOSAL:

October 21, 1996

# OVERALL SUMMARY FOR THIS REPORTING PERIOD

The body of the miniature long path gas cell arrived from the machine shop. The body has been supported on the optical bench, along with transfer optics and the FT-IR spectrometer. The end-cap mirrors are all that are needed to complete the Phase I system. Diamond machining of the end-cap mirrors has been completed, and the parts were sent to the vendor for coating with protected silver.

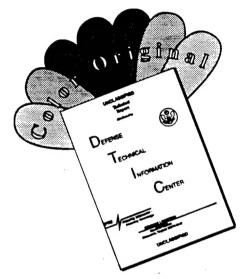
Measurements were made for trichloroethylene (TCE) with a gas analyzer equipped with a 20 meter, 1.6 liter volume cell that used gold coated mirrors of less than optimal surface quality. 50 ppb TCE was easily detected; 20 ppb TCE did not greatly emerge above the instrumental baseline noise level. The results were promising when analyzed with regard to that expected for the novel cell being constructed.

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# TASK 1 - DESIGN AND CONSTRUCT MINIATURE LONG PATH GAS CELL

Objective: To develop a multi-pass gas cell with maximum pathlength and minimum volume employing On-Line's patented non-spherical, aberration-correcting optics.

Summary of Progress: The gas cell body of the miniature long path cell arrived from the machine shop. Photos of the open assembly (body and cover) are shown in Fig. 1. The tapered shape is seen which minimizes sample volume outside of the multipassed infrared beam. The machined ridges along the wall of the taper are "light baffles", to inhibit stray light from reflecting from otherwise smooth walls in directions nominally the same as the main beam.

The closed assembly (less end-cap mirrors) is shown in Fig.2. Observed are the openings for the end-cap mirrors and KBr window. The end-cap mirrors are necessary to complete the system.

Diamond machining of the end-cap mirrors was completed on Sept. 3, 1996. The parts have since been sent to the next vendor for coating with the highly reflective protected silver. Delivery of the completed mirrors to AFR is expected between Sept. 13 - 20, 1996.

Plans and Objective for Next Reporting Period: Upon arrival of the end-cap mirrors, full assembly of the gas cell will be completed. Connections will be made for gas flow through the cell, and optical coupling to the FT-IR gas analyzer will proceed (see Task 2).

# TASK 2 - COUPLE MINIATURE GAS CELL TO ADVANCED FT-IR GAS ANALYZER

Objective: To design, purchase and implement the infrared transfer optics to couple the miniature gas cell to a contractor owned FT-IR spectrometer suitable for on-site use.

Summary of Progress: Figure 3 presents photographs of the cell body (less end-cap mirrors) supported on the optical bench with transfer optics and the FT-IR spectrometer. Also seen is a HeNe laser which provides a red (visible) beam which will be along the same path as the IR beam, for alignment purposes. The end-cap mirrors are needed to complete the optical system and proceed with beam alignment through the system.

Plans and Objective for Next Reporting Period: Complete assembly and optical alignment.

# TASK 3 - TEST SYSTEM RESPONSE AND DETECTION LIMITS TO TRICHLOROETHYLENE CONTAMINATION

Objective: The Phase I monitor will be demonstrated for measurement sensitivity and speed during TCE monitoring.

Summary of Progress: Work in this task focused on performing preliminary measurements on gas samples containing low concentrations of TCE. To do this, a standard "On-Line Multi-Gas 2001" FT-IR analyzer was utilized, which had a 20 meter pathlength, 1.6 liter volume gas cell. The mirrors of this gas cell have been exposed to several corrosive gas streams in the past, and

also have been in contact with hot water droplets from a sample line containing condensed water. The gold coated surfaces of the mirrors were observed to be somewhat pitted, a less than ideal condition that would decrease infrared throughput and result in a decreased signal-to-noise ratio.

Figure 4 presents measured spectra for TCE in nitrogen at concentrations of 108 ppb (parts per billion), 54 ppb, and 18 ppb. These concentrations were obtained by syringe injection of calibration gas (9.8 ppm TCE) into the 1.6 liter volume. The spectra were collected with an instrument resolution of 1 cm<sup>-1</sup>, and 1000 individual scans were co-added in just under 5 minutes for each spectrum. (Figure 5 shows the spectrum of TCE supplied in the Hanst library of FT-IR spectra, at a much higher concentration (282 parts per million per meter), for comparison.

The observation of Figure 4 is that the three main absorption bands of TCE extend well above the baseline noise even at a concentration of 54 ppb. At 18 ppb, the absorption bands are just within the noise. The results are promising, however, given the condition of the mirrors in this instrument. The results do not go against our premise that single digit ppb sensitivity will be achieved with the low volume 50 meter cell utilizing the aberration correcting mirrors of highly reflective protected silver.

Plans and Objective for Next Reporting Period: Begin measurements of TCE in the 50 meter, 500 cc volume cell once Task 2 is completed.

# TASK 4 - PLAN FOR PHASE II PROTOTYPE

Objective: To evaluate the degree to which the overall Phase I objective was met and to formulate a preliminary design for a Phase II prototype

Summary of Progress: No work performed under this task.

# MEETINGS AND/OR IMPORTANT TELEPHONE DISCUSSIONS

- 1) Due to the long delivery time of the gas cell mirrors, a request for a six week no-cost extension to the program was submitted to the contracting officer. The extension was granted, extending the period of performance to Nov. 29, 1996. In consideration for the time extension, the contractor will provide to the Government a draft final report for review and comment by Nov. 15, 1996. If revision is necessary, the final delivery date is Nov. 29, 1996.
- 2) The contractor has been notified that the Phase II proposal due date is Oct. 21, 1996.
- 3) Testing of the Phase I apparatus at Armstrong Laboratory has been tentatively scheduled for the week of Sept. 30 Oct. 4, 1996. It is planned to confirm this schedule in the first week of the next reporting period.

# STATUS OF PROPOSED WORK SCHEDULE

5111165 51 1165		Months					
		May	June		Aug	Sept	Oct
	0	1	2	3	4	5	6
Task 1 - Design and Construct Miniature Long Path Gas Cell		1		2			
Task 2 - Couple Miniature Cell to Advanced FT-IR Gas Analyzer			3	3	4		
Task 3 - Test System Response to Trichloroethylene Contamination						5	
Task 4 - Plan for Phase II Prototype							6

# **Anticipated Milestones**

- 1 Test and design completed
- 2 Cell fabricated
- 3 Coupling optics ordered

- 4 System completed
- 5 TCE tests completed
- 6 Analysis completed

With the Phase I contract extension, the Oct. 21 due date of the Phase II proposal is the key deadline for demonstration of measurement feasibility. If the gas cell mirrors are delivered within the present anticipated time frame, the objectives should be met.

James R. Markham

Principal Investigator

Chad M. Nelson

Program Manager

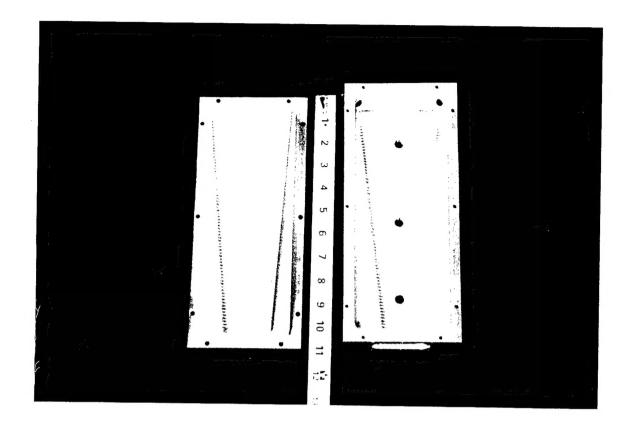
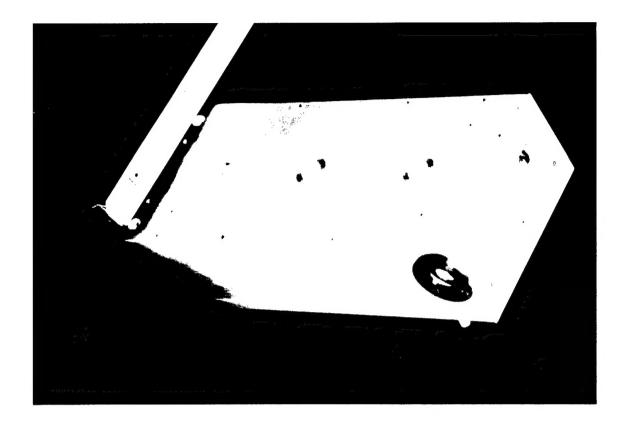




Figure 1. Photographs of Open Gas Cell Body (right) and Cover (left).



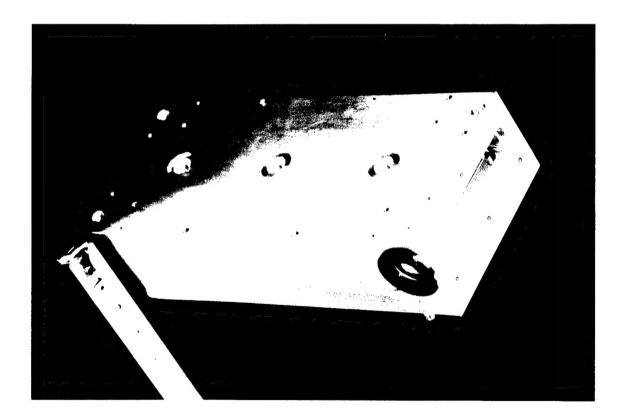


Figure 2. Photographs of Gas Cell (less end-cap mirrors). The Black Retaining Ring Holds the KBr Window in Place Near the End which will Hold the Field Mirror.

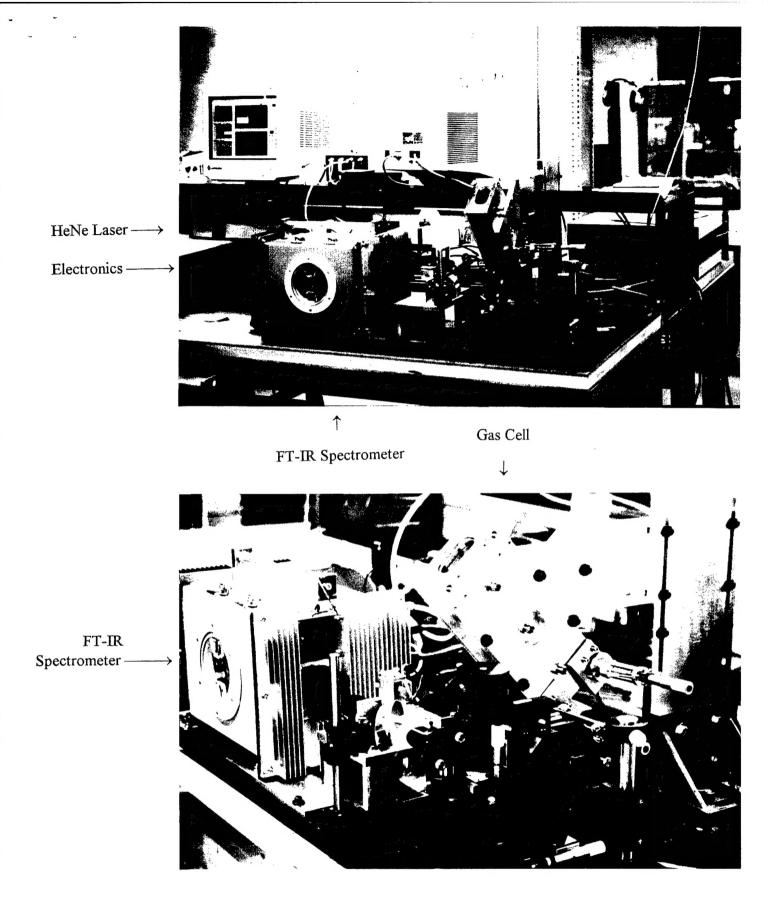
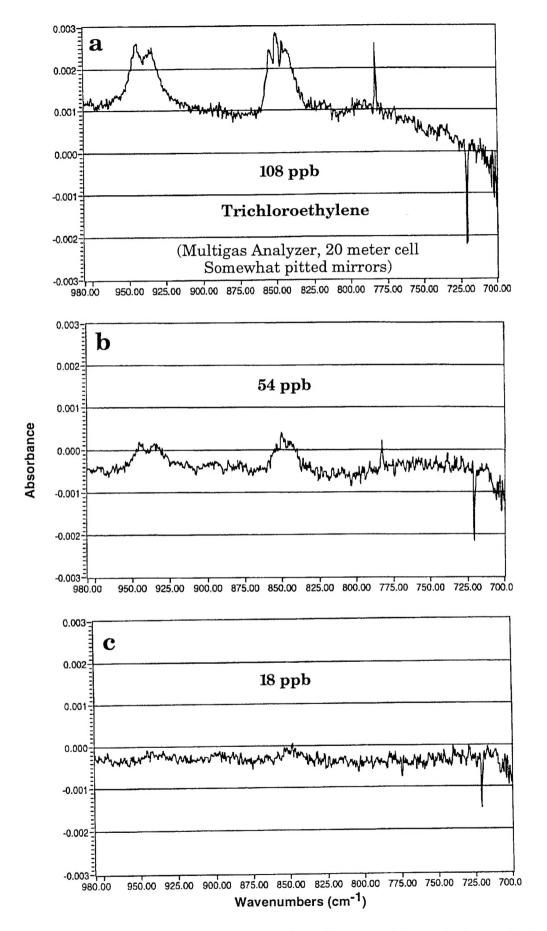


Figure 3. Photographs of Phase I Optical Assembly (less end-cap mirrors on gas cell). The Black Optical Platform with the Many Tapped Holes is 36" x 24" x ½" Thick. The Opening on the Gas Cell for the Objective Mirror is Seen.



**Figure 4.** Spectra of Trichloroethylene Measured in a 20 Meter,  $1.6 \,\ell$  Gas Cell at Three Concentrations.  $1 \, \mathrm{cm}^{-1}$  Resolution, 1000 Scans in 4 min. 50 sec.

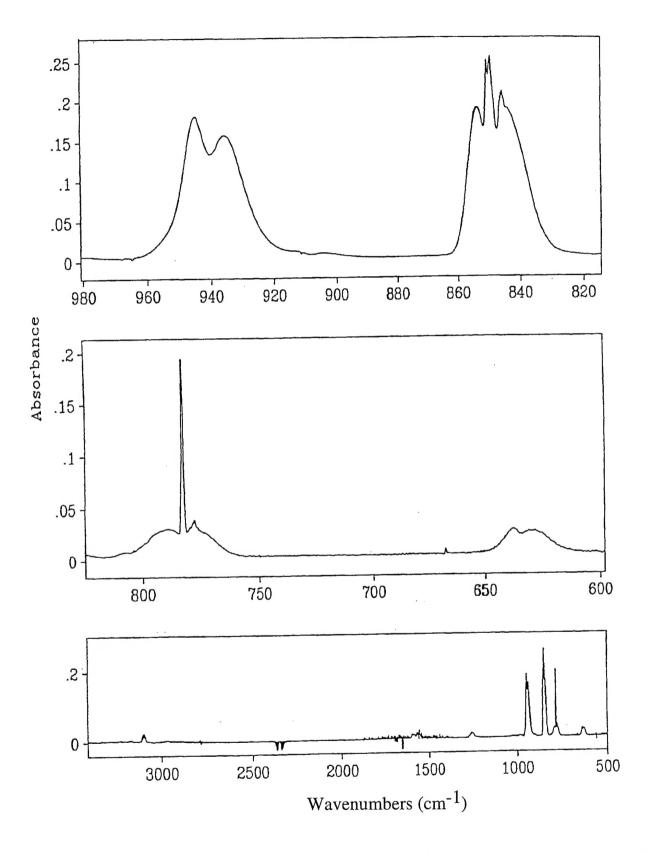


Figure 5. Hanst Library Spectrum of Trichloroethylene, 282 micro-atm Meters, in 1 atm  $\rm N_2$ , 0.5 cm  $^{-1}$  Resolution.